IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	)	Art Unit: JACQUINOT=7
Eric JACQUINOT et al	)	Examiner: Duy DEO
Appln. No.: 09/427,675	)	Washington, D.C.
Date Filed: October 27, 1999	)	Confirmation No. 3607
For: NEW ABRASIVE COMPOSITION FOR THE INTEGRATED…	)	ATTY.'S DOCKET:

DECLARATION UNDER 37 CFR 1.132

Honorable Commissioner for Patents
Washington, D.C. 20231

Sir:

The undersigned Eric JACQUINOT hereby solemnly declares as follows:

That he is a French Citizen living in: TROSLY BREUIL (France);

That he is graduated PhD in Inorganic Chemistry (study of catalytic properties of modified zeolites);

That since 1989, he has been employed by Clariant (France) (formerly named "Société Française Hoechst");

That he is co-inventor in eight (8) U.S. patents, several of which relate to chemical mechanical polishing, and one of which is the main reference relied upon by the U.S. Examiner, namely Jacquinot USP 6,043,159;

That he is a (co)inventor of the present invention and an applicant of the above-identified U.S. patent application;

That he has read and is familiar with the above identified application, the official actions and the prior art;

That he considers himself expert in the present art by his knowledge of Chemistry and especially by his 16 years experience in the field of silicate and silica sol chemistry;

That the following comparative tests were conducted under his direction in order to compare the results of the present invention with the results obtained by following Jacquinot et al USP 6,043,159 and Grover et al USP 5,759,917.

Method of chemically mechanically polishing (CMP):

The CMP slurries were used to chemically mechanically polish blanket silicon oxide and silicon nitride using a IC 1000 pad manufactured by Rodel. The polishing was performed using a

Alpsitec MECAPOL E 460 polisher at a down force of 7 psi, a slurry flow rate of 100 ml/min, a platen speed of 30 rpm and a carrier speed of 42 rpm. Except for the CMP slurries, all conditions remained the same in all the tests.

Colloidal Silica formulations:

A silica slurry, composed of varying weight percent amounts of colloidal silica, pH approximately 2.5, manufactured by Clariant (France), was formulated as shown in the table below. The pH of the slurry for slurries 4 and 5 was adjusted to about 4.2 by adding commercial ammonium hydroxide.

Precipitated Ceria Nitrate formulations:

A nitrate stabilized ceria slurry, pH 1.8 and 20 % solids, was purchased from Nyacol Products (SPCI S.A.). The pH of slurries 8 and 9 was adjusted to about 4.2 by adding highly diluted[1] ammonium hydroxide. The polishing results using the different CMP slurries are reported in table 1 below.

[1] When commecial ammonium hydroxide (32%) was used, precipitation occurred immediately. Several dilutions were tested, but even with a 1% ammonium hydroxide, precipitation occurred immediately. Consequently, only a 0.5% dilution allowed the preparation of slurries 8 and 9.

Table 1

Slurry N°	Slurry type	Example	Abrasive type	pH	Abrasive %(weight)	Surfactant %(volume) ®Emulsogen EP	TEOS RR (Å/min)	Si_3N_4 RR (Å/min)	Selectivity
1	Jacquinot Prior art USP 6,043,159	Test 9 comparative	colloidal silica	2.5	30	0	1905	438	4.4
2	present invention	Test 8 present invention	colloidal silica	2.5	30	1	2075	4	520
3	present invention	Test 7 present invention	colloidal silica	2.5	30	0.5	2193	7	313
4	Jacquinot USP 6,043,159	New example	colloidal silica	4.2	20	0	775	735	1.05
5	present invention	New example	colloidal silica	4.2	20	1	816	16	51
6	Grover like		precipitated ceria nitrate	1.8	20	0	75	4	18
7	Grover like		precipitated ceria nitrate	1.8	20	1	56	2	28
8	Grover USP 5,759,917	Example 3 Grover	precipitated ceria nitrate	4.2	15[2]	0	245	5	50
9	modified Grover USP 5,759,917	Example 3 Grover	precipitated ceria nitrate	4.2	15[2]	1	270	2	135

Tests 6 and 7 confirm the teachings of Grover that a

[2] Because of the significant amount of water provided by the 0.5% ammonium hydroxide, see footnote 1 on page 3 above, we were able to provide an abrasive % of only 15% for slurries 8 and 9.

weak polishing (TEOS results) is obtained at pH 1.8. Therefore from the teachings of Grover and the comparison among tests 6-9, and particularly comparing tests 6 and 8 wherein no surfactant is used consistent with the preferred operation of Grover, the skilled person learns that a low pH should not be used, and in my opinion the skilled worker seeking to learn from Grover would avoid using a low pH.

Comparing tests 7 and 9, which are like tests 6 and 8 respectively except that the slurries 7 and 9 contain surfactant, one sees that better results are obtained at the higher pH, thus confirming what is taught by Grover and what is also learned from the above-noted comparison between tests 6 and 8.

Next, comparing tests 8 and 9, one sees that there is little difference between the presence of surfactant (slurry 9) and the absence of surfactant (slurry 8). The TEOS rate is only slightly better with the presence of surfactant. The difference in polishing speed of Si_3N_4 between the two tests is not a reliable indicator of the advantage of either one because both values are so small that accurate measurements are difficult at best, and the decrease to 2 Å/min in test 9 (from the value of 5 in test 8) provides no evidence of any increase in selectivity.

From the comparison between tests 8 and 9, the skilled worker learns that there is little difference and that no substantial advantage can be achieved by adding surfactant to Example 3 of Grover.

In slurry N° 9, the inclusion of surfactant provides a selectivity of 135 to be compared with the selectivity value of 50 for slurry N° 8 (factor increase of 2.7), which is not a large factor increase. On the other hand, a comparison between the tests 1 and 2 evidences an increase by a factor of 119, and a comparison between tests 4 and 5 (new example 5 is within the scope of the claims) evidences a selectivity increase by a factor of 49.

The tests 8 and 9 evidence that a better speed of polishing is obtained than in tests 6 and 7, respectively, but this speed is nevertheless low in comparison with the speed of polishing obtained according to the teachings of the present invention. These comparative results teach that the addition of surfactant (slurries 2, 3 and 5) to Jacquinot prior art (slurries 1 and 4) produce immense increases in selectivity, far greater than what could have been predicted from the comparison between the selectivity results of slurries 8 and 9.

Further, a comparison between the tests 1 and 2 shows that at a pH of 2.5 the initial speed of polishing of Si_3N_4

dramatically and unexpectedly decreases from 438 Å/min to 4 Å/min, i.e. a factor of more than 100. An excellent decrease by a factor of about 46 is also observed between tests 4 and 5 at pH 4.2.

The above comparisons prove that the present invention provides not only far superior results compared to Jacquinot '159, but also far better results than Grover either as shown (slurry 8) or as modified (slurry 9).

Stability results

A chemical mechanical slurry is said to be stable if it keeps its colloidal properties (absence of agregates and/or sedimentation) without agitation and when a sampling is made from any part of the slurry.

The stability results are reported in the table 2 below.

Table 2

Slurry N°	Slurry type	Stability
1	Prior art Jacquinot USP 6,043,159	At least 3 months and generally 6 months
2	present invention	At least 3 months and generally 6 months
3	present invention	At least 3 months and generally 6 months
4	Jacquinot USP 6,043,159	At least 3 months and generally 6 months
5	present invention	At least 3 months and generally 6 months
6	Grover-like	At least 3 months and generally 6 months
7	Grover-like	At least 3 months and generally 6 months
8	Grover	Precipitation after 13 days
9	modified Grover	Precipitation after 13 days

Table 2, slurry 8, discloses a defect in the Grover system, i.e. lack of stability. Slurry N° 9, just like slurry 8 except for the addition of surfactant, provided no improvement over slurry 8. From a comparison of tests 8 and 9, the person skilled in the art learns that there is no advantage to be gained insofar as stability is concerned by adding surfactant to the Grover example (test 8).

Conclusions

1. Insofar as stability is concerned, it is surprising that the slurry of the present invention remains stable at a pH of 4.2 as well as at a pH of 2.5, whereas when

the pH of the Grover slurry is at 4.2, it is unstable, precipitates quickly and therefore may not be stored before usage. The present invention therefore provides an important advantage over Grover with respect to pH's in the neighborhood of 4.2. yet a higher pH is necessary in the Grover system (comparing tests 6 and 7 with tests 8 and 9) in order to provide reasonably good abrasion results.

2. The addition of surfactant to the Grover slurry provides no advantage insofar as stability is concerned. The addition of surfactant to the Grover slurry (slurry 9 compared with slurry 8) and the addition of surfactant to a Grover-like slurry (compare slurry 7 with slurry 6) provides no significant advantage with respect to polishing results. Therefore, no meaningful incentive exists to add surfactant to the slurry of Grover.

3. Consistent with the teachings of the present invention, very great advantages are achieved by adding surfactant to the slurry of Jacquinot '145. These results are very surprising.

4. The results of the present invention are also dramatically improved over Grover (slurry 8), over modified Grover with surfactant (slurry 9) and over Grover-like slurries 6 and 7.

In re of Appln. No. 09/427,675

I hereby further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Eric Jacquinot

Eric Jacquinot

Date: JULY 09, 2003

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